Application Serial No.: 09/817,567
Attorney Docket No. 11299-006-999

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:
SUBRAMANIAN et al.

Confirmation No. 9915

Serial No.: 09/817,567

Examiner: Daniel J. Davis

Filed: March 26, 2001

Group Art Unit: 3731

Title: SILICON MICROLANCET DEVICE AND METHOD OF CONSTRUCTION

Attorney Docket No.: 11299-006-999

DECLARATION UNDER 37 CFR 1.131

I, Wilson Smart, declare and state that:

1. I am one of the inventors of the above identified patent application.
2. I am the President and Chairman of the Board of Kumetrix, Inc., the assignee of the invention that is the subject of the above-identified application.
3. The present invention was reduced to practice in the United States prior to the June 9, 1999, filing date of U.S. Patent No. 6,379,324 B1.
4. This reduction to practice is evidenced by the attached excerpt (Exhibit A) from a Special Technical Report submitted to the Defense Advanced Research Projects Agency pursuant to Contract DAAH 01-95-C-R118. The date of this Report, which has been deleted, is prior to 1999. Several other dates have also been removed from this Report, all of which are prior to 1999.
5. As set forth on page 8 of the Report, silicon cantilevers or needles have been produced that are 50μm wide x 50μm thick x 3 mm long. As shown in Fig. 3 on page 8 the needle portion extends from a substantially larger portion. As described on page 8, the needle is produced from a single silicon wafer that is thinned with hot KOH. Free-standing needles are then produced by etching the wafers through with a plasma.

6. Thus, with reference to claim 24 of the above-identified application, attached page 8 describes a silicon needle formed from a single silicon substrate and having a base portion and a needle portion extending therefrom that can be used for penetration. Further, the dimensions of the needle portion are 50μm wide x 50μm thick x 3 mm long.

7. Further evidence of the reduction to practice is set forth in the attached excerpt (Exhibit B) from a second Special Technical Report submitted to DARPA pursuant to Contract DAAH-95-C-R118. The date of this second Report, which has been deleted, is later than the Report that is attached as Exhibit A but is prior to 1999. Several other dates have also been removed from this Report, all of which are prior to 1999.

8. As set forth on page 5 of the second Report, eight different needle designs were fabricated for experimental evaluation of needle strength. As shown in Fig. 1 on page 5, several of the experimental needle geometries varied in width and shape. The needles were then subjected to fracture testing, and the average critical force and average critical displacement at fracture measured. Finite element analysis of the best-performing needles was then used to further improve needle design. The needle that is pictured in the bottom left hand corner of Figure 1 is identified elsewhere in the report as "Column 2" and the needle pictured in the bottom center of Figure 1 is identified elsewhere as "Column 3." Masks for these two needles are depicted in Figures 10 and 13, respectively. These masks give feature dimensions in millimeters. Of particular note, the needle widths range from about 0.05 millimeters to 0.3 millimeters in the case of the Column 2 needle and 0.05 millimeters in the case of the Column 3 needle. Based on stress tests, an improved hybrid lancet was designed with a base width and taper angle intermediate between those shown in the bottom left and bottom center photographs of Fig. 1 and a slightly shorter, wider tip than in the bottom center design.

9. I declare further that all of the statements herein of my own knowledge are true; and all statements made on knowledge and belief are believed true, and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the present application and any patent issuing thereon.

Date: April 29, 2004

Wilson Smart

SPECIAL TECHNICAL REPORT

INTEGRATED SAMPLING DEVICE FOR MICROFLUIDIC BIOANALYSIS SYSTEMS

Sponsored by

Defense Advanced Research Projects Agency (DOD)

DARPA Order No. D611, Amdt. 09

Issued by U.S. Army Missile Command Under

Contract #DAAH01-96-C-R118

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Effective Date of Contract:

Short Title of Work:
Microfluidic Bioanalysis

Work Expiration Date:

Reporting Period:

NEEDLES

Silicon cantilevers 50 μm wide x 50 μm thick x 3mm long were produced using the methods described in our previous report. Thinning of the silicon to establish the needle thickness dimension was done with hot KOH, and etching through the wafer to produce free standing needles was done with plasma. These "needles" (solid cantilevers) were produced from a single silicon wafer and they did not have a bore. The purpose of this experiment was to show that silicon etching methods are indeed superior to glass, and that the mask set and processing methods that we are using are capable of yielding precise silicon needles with cross sections less than 100 μm. A test device with a 50 μm "needle" is shown in Figure 3.

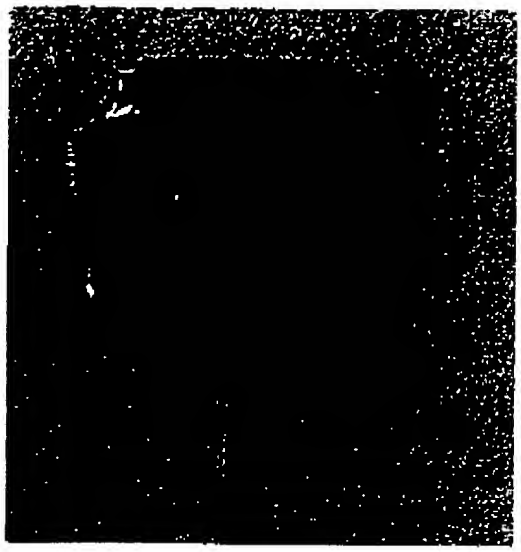

Figure 3 Experimental 50 μm Silicon Needle

The technique that produced the device of Figure 3 should be able to produce actual all-silicon needles (with a bore) as part of microsampling and assay devices. The only difference will be that the latter will be made from two silicon wafers bonded together rather than from a single wafer. Depending on the wafer bonding method that emerges from those experiments, described below, there might be a thin film of a bonding agent (metal alloy, glass, polymer) at the interface between the two wafers which the needle etch step will have to etch through. This bonding material is expected to be sufficiently thin (~1 μm) that it will be able to be removed in a practical process. The strategy for external etching to form the needles is to thin the silicon on the top and bottom of the needles with KOH and then etch through between the needles with plasma.

We feel that the production of all-silicon needles in the size range desired is feasible using methods now in hand.

SEMI-ANNUAL TECHNICAL REPORT

INTEGRATED SAMPLING DEVICE FOR MICROFLUIDIC BIOANALYSIS SYSTEMS

Sponsored by

Defense Advanced Research Projects Agency (DOD)
Electronics Technology Office

DARPA Order No.E612, Amdt. 17

Issued by U.S. Army Missile Command Under

Contract #DAAH01-97-C-R113

Name of Contractor:
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Effective Date of Contract:

Contract Expiration Date:

Short Title of Work:
Microfluidic Bioanalysis

Reporting Period:

REPORT DOCUMENTATION PAGE (SF298)

1. AGENCY USE ONLY (leave blank)

2.REPORT DATE

3. REPORT TYPE AND DATES COVERED
Semi-Annual Technical Report,

4. TITLE AND SUBTITLE
Integrated Sampling Device for
Microfluidic Bioanalysis Systems

5. FUNDING NUMBERS
Contract No. DAAH01-97-C-R113

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11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
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Attn: Technical Information
Ms. Debra Amick, DARPA

12B. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

Design, fabrication, and evaluation of silicon micro-needles was the principal focus of the work of this period. Information and advice from our entomology, dermatology, and engineering consultants guided our selection of eight needle designs for testing and stress calculations. An apparatus was built to measure the force and displacement as the tip of the needle was displaced until needle fracture occurred. Three of these designs show promise for application in painless blood testing as a result of these experimental fracture measurements and theoretical analyses. Besides the needle development, the silicon nitride cuvette windows were improved and optically characterized. Experimental work on the glucose assay in whole blood using glucose dehydrogenase and MTT tetrazolium salt indicates that this method is likely to be practical for our consumables. Demonstration that a deposited polysilicon thin film on silicon nitride permits anodic bonding of a glass wafer to a silicon wafer incorporating silicon nitride windows has provided encouragement that anodic bonding using multilayer films will be successful for silicon-to-silicon wafer bonding in these biodiagnostic consumables.

14. SUBJECT TERMS
MEMS
Microfluidics
Bioassay
Microelectromechanical

15. NUMBER OF PAGES
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16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	UL

TASK 1: FABRICATION OF EXPERIMENTAL CONSUMABLES

A mask was made incorporating eight different needle geometries and dimensions, six of which are shown in Figure 1. This mask was used to create "needles" (silicon cantilevers without bore) for experimental evaluation of needle strength in Task 2.2, below. Although the processing steps from wafer to wafer were nominally the same, experience with processing details, particularly the final etching to produce freestanding needles, led to an improvement in surface finish with later wafers.

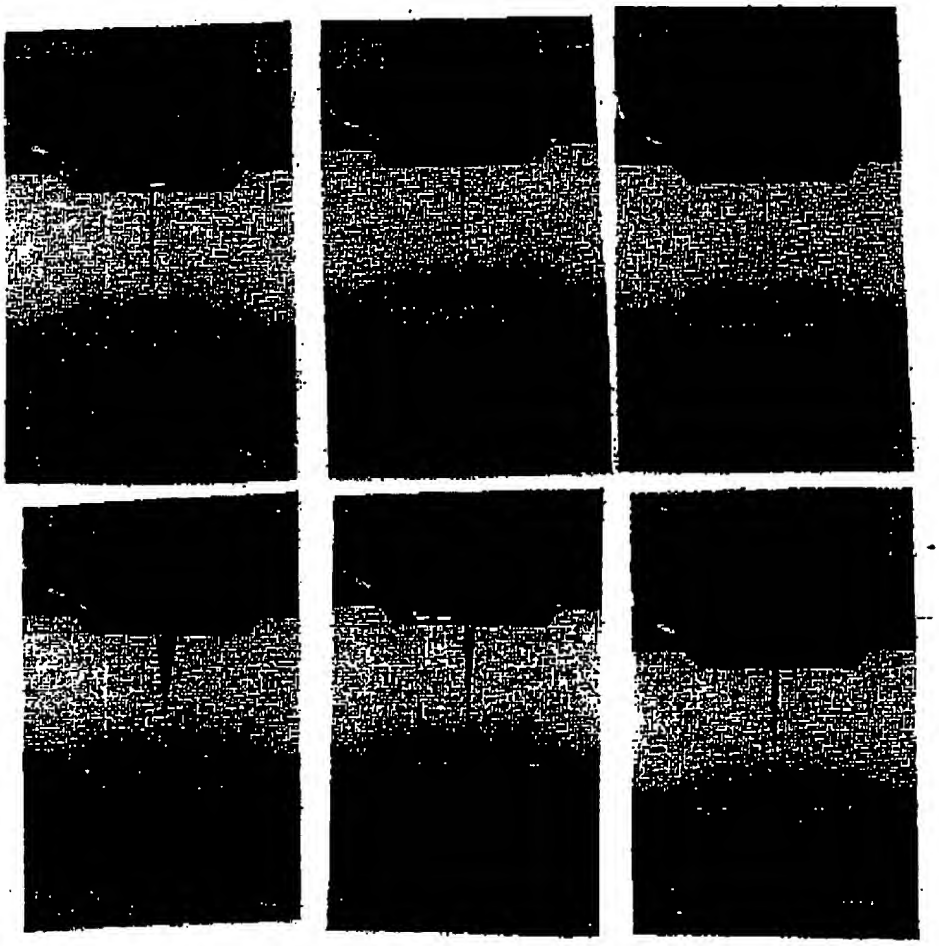


Figure 1 Needle Geometries for Fracture Testing

THEORETICAL ANALYSIS AND NEEDLE STRENGTH MEASUREMENTS

Abstract:

Physical testing and finite element simulations of the fabricated silicon needles are being performed in order to establish the validity of the numerical modeling and to determine the strength of the needles for wafers ranging in thickness from 60-70 μm. Several different needle geometries were tested in both the in-plane and out-of-plane orientations. Early test results showed the out-of-plane to be the weakest, as expected, and this orientation is focused upon. Similarly, various needle geometries were found early to be too weak for testing and were abandoned. The physical testing of applied force required to break the needles agrees closely with the theoretical analysis, while two of the maximum physical displacements are larger than predicted values. Further computer modeling and physical testing is being performed to identify the cause of this inconsistency. The experimental and theoretical values are shown below:

Experimental		Theoretical
Average Critical Force (N):		
Column 2:	6.71E-02 ± 7.72E-03	6.10E-02
Column 3:	1.44E-01 ± 1.08E-02	1.18E-01
Column 5:	4.08E-02 ± 4.61E-03	4.30E-02
Column 6:	3.66E-02 ± 4.55E-03	4.30E-02
Column 8:	4.13E-02 ± 5.81E-03	3.80E-02
Average Critical Displacement (mm):		
Column 2:	3.99E-01 ± 1.81E-01	2.50E-01
Column 3:	4.75E-01 ± 2.63E-01	2.00E-01
Column 5:	4.04E-01 ± 1.67E-01	2.40E-01
Column 6:	3.36E-01 ± 1.97E-01	2.60E-01
Column 8:	4.97E-01 ± 1.27E-01	2.00E-01

Introduction:

The physical testing of silicon needle strength is a fundamental step in the process of developing a painless blood-sampling device. The purpose of this stage of development is to investigate the various needle designs proposed as potential prototypes. An understanding of the forces and displacements that the current needles can withstand will allow the research team to identify the most promising designs and to make the necessary changes to optimize needle strength, yet remain on a scale that will prove painless to the user. Another factor that needs to be considered in the physical testing is the location of the fracture. It is not only important to note the forces at which the needles break, but also where the fractures occur is equally important for effecting design modifications.

Silicon cantilevers without a bore were used for the experimental testing and also as models for the calculations. Fracture data for these cantilevers is valid for actual needles (with bore) because the bending stiffness of a needle varies according to the FOURTH power of the characteristic length of the cross-section. The stiffness of the beam with the bore's dimensions has to subtracted to account for the bore. More precisely, the stiffness is the moment of inertia, I, where

accurately and efficiently. A large-deformation analysis is performed to account for nonlinear geometry effects.

The meshes are refined in regions where high stress is expected, and each mesh extends a distance of 50μ into the bulk of the wafer from the base of the needle itself. The nodes of this extreme face of the mesh are fixed in all directions to simulate the boundary conditions of the test fixture. A displacement out of the plane of the wafer is then applied to a node near the tip of the needle, at a point as close as possible to the application of the load in the experiments. The stresses predicted by the FEA code are examined to establish the location where the fracture is expected to initiate. The maximum principal stress in any direction at the surface of the needle is chosen as the failure criterion, since it is assumed that microcracks exist at arbitrary orientations throughout the material.

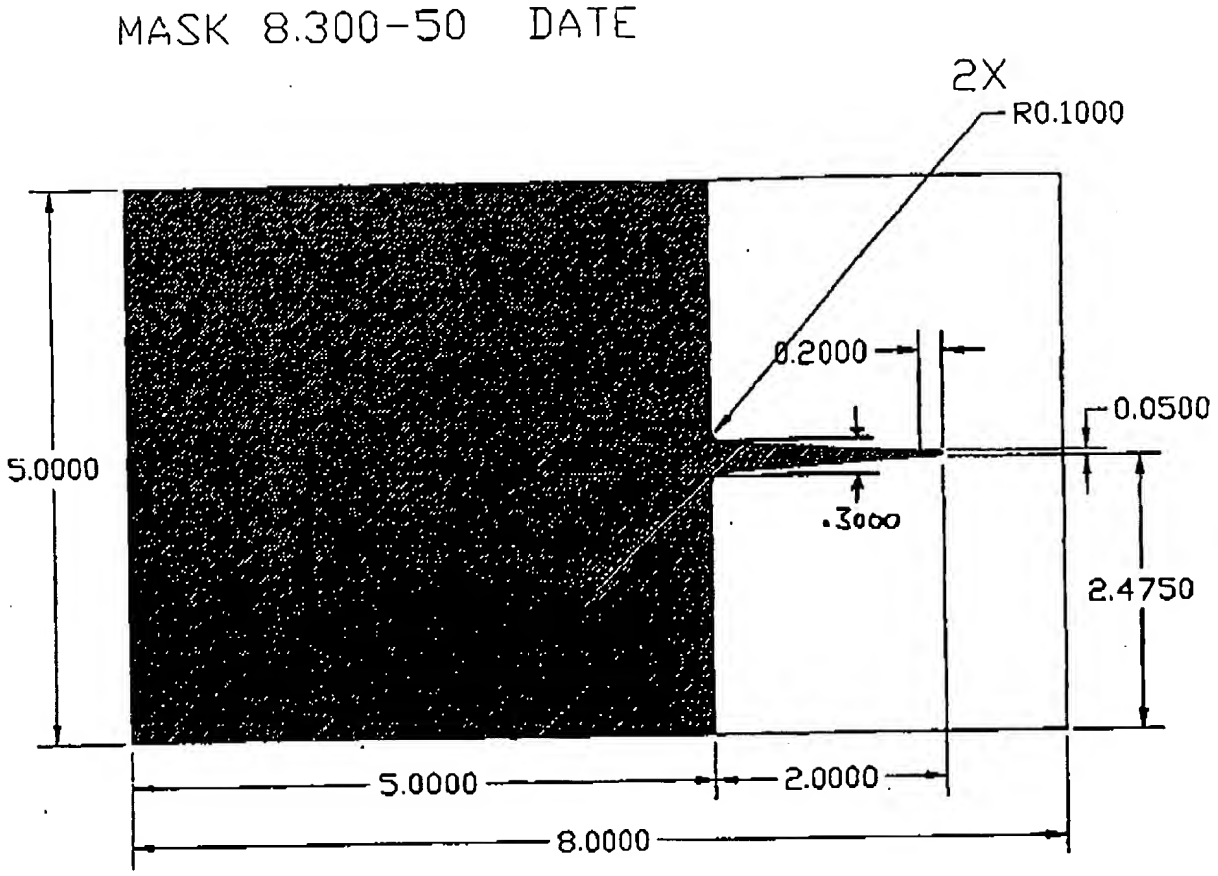
Guided by the strength values for single-crystal silicon given in the literature and by initial experimentation, the needle is deflected until a maximum principal stress of 700 Mpa is predicted by the FEA code at some point on the surface. This critical deflection and the reaction force required to produce it are then compared with the empirical data.

It is seen that the geometries associated with columns 1, 4, and 7 have been predicted to have a reaction force substantially lower than the more robust designs. This can be expected due to the small widths and straightness of the designs, while the wider-based and tapered needles are reasonably stronger. The tapered nature of the other designs allows for a better distribution of stress throughout the needle. In the straighter needles the stresses increase quickly at their bases, resulting in a fracture for far smaller values of needle deflection. In the tapered needles the highest stress area is still at the base, however the taper allows the stress to be better transmitted into the adjacent areas resulting in a greater deflection before the critical reaction force is reached.

Quantitatively, we may state that the stress goes as $(\text{width})^{-1}(\text{thickness})^{2}$. Therefore, as the base width is increased, as in the tapered designs, the stress value decreases for a specific deflection. This results in a greater force being required to reach the critical stress fracture value.

Below are shown diagrams of some of the needle parameters and finite element meshes:

Figure 10



Column #2

Figure 11

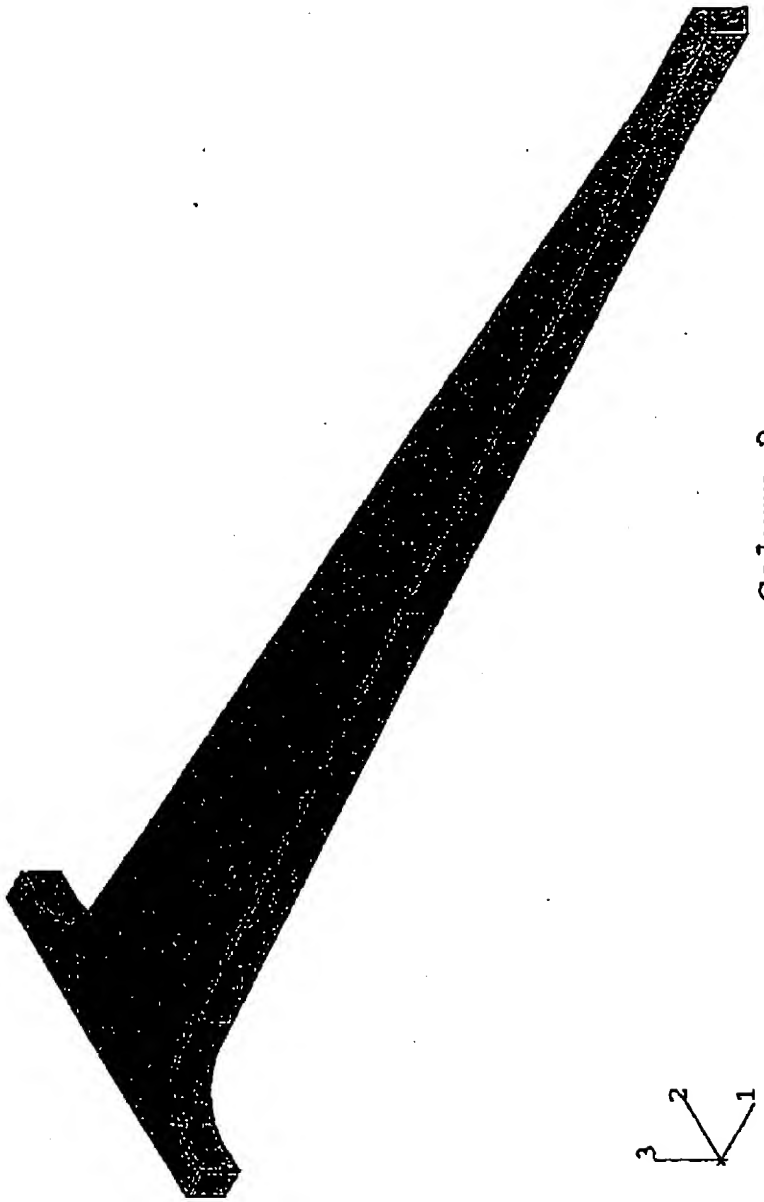


Figure 12

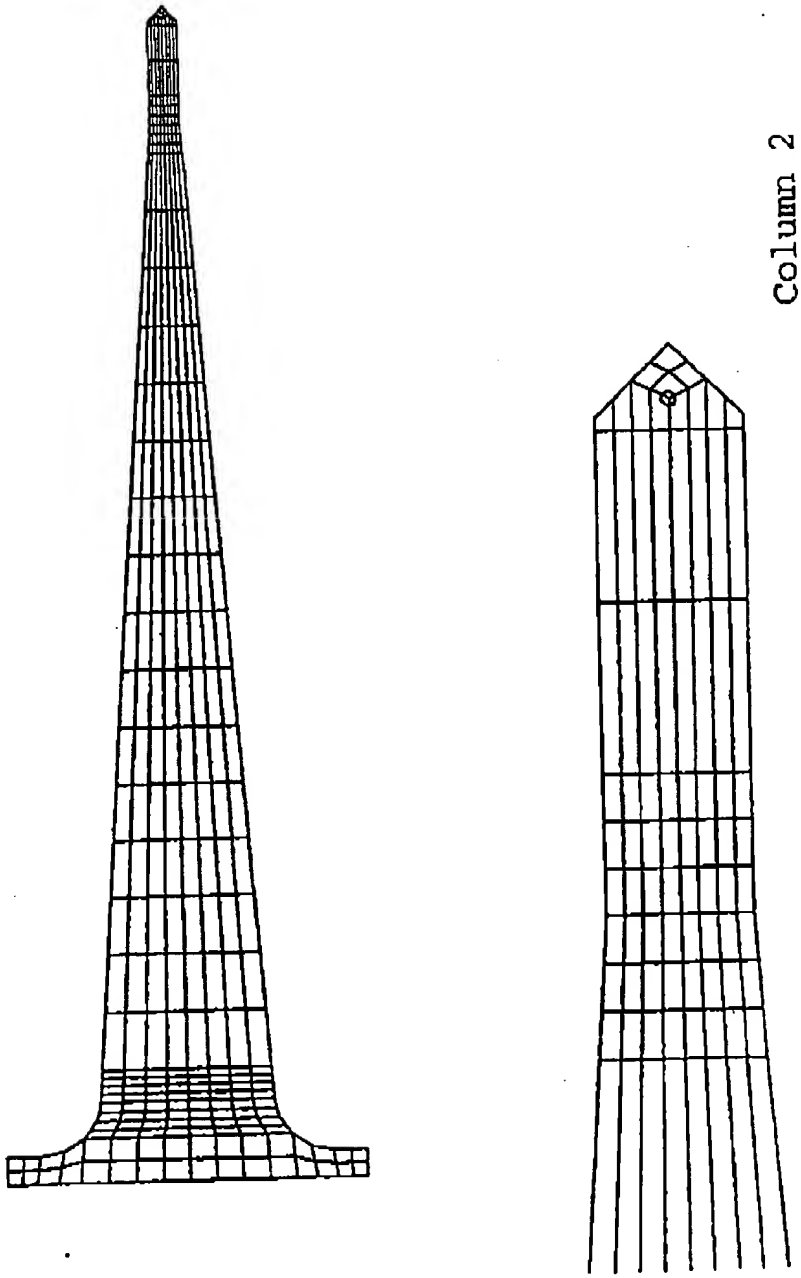
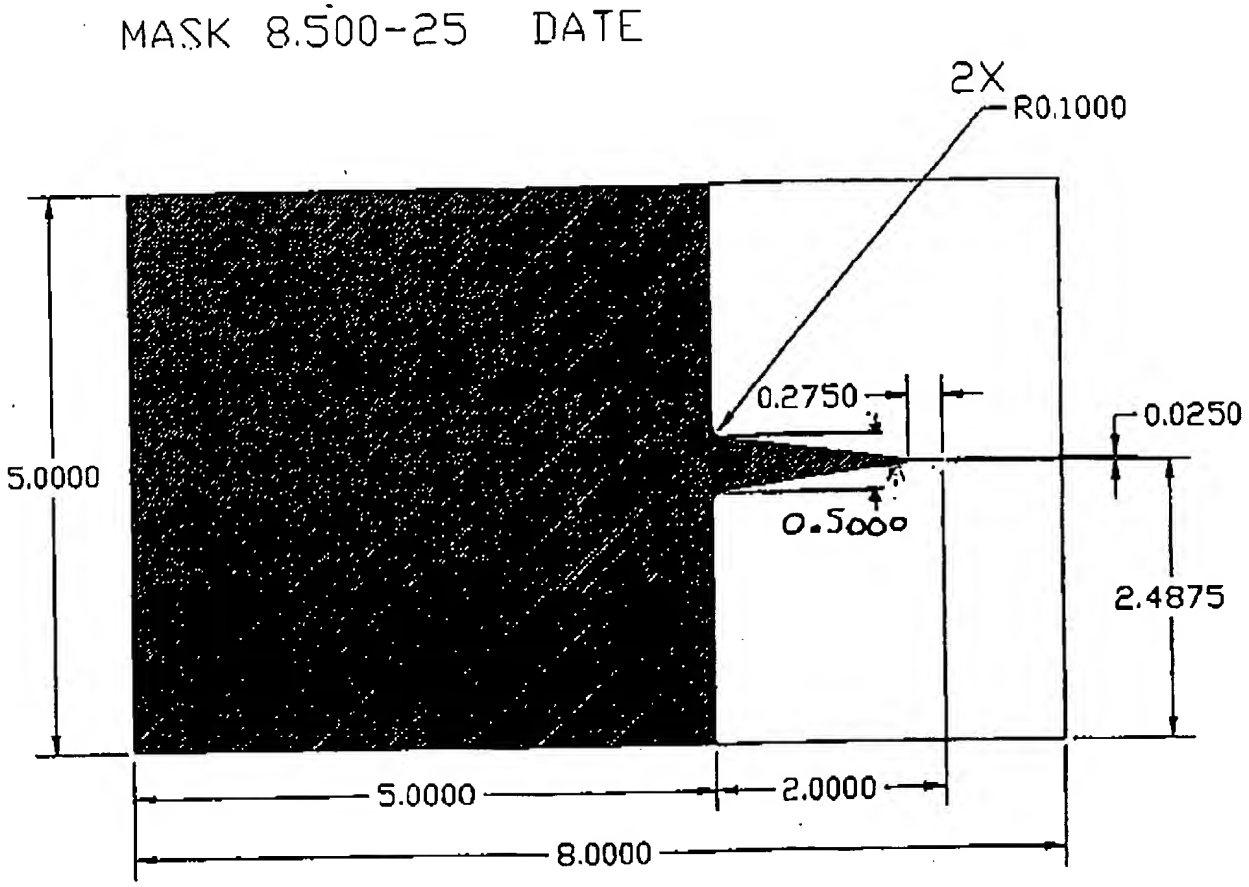


Figure 13



Column #3

Figure 14